REMARKS

This Amendment and Response is responsive to the Examiner's July 31, 2009 Office Action. After entry of the present amendments Claims 1-4 will be pending. Claim 21 is canceled herein.

Rejections under 35 U.S.C. § 112

Claim 21 was rejected by the Examiner under 35 U.S.C. § 112, first paragraph because the best mode contemplated by the inventor has not been disclosed. Applicant notes that the portion of the specification refers to condensing to a liquid film on the substrate surface. Thus, Applicant respectfully disagrees with the Examiner's interpretation of the language of previously pending Claim 21. Without acquiescing in the rejection, Claim 21 is canceled to speed prosecution.

Rejections under 35 U.S.C. § 103

Claims 1-4 stand rejected by the Examiner under 35 U.S.C. § 103 as obvious in light of Dillon et al., Surface Science 322 (1995), 230-242 ("Dillon"), in view of Penneck et al., U.S. Pat. No. 4,985,313 ("Penneck").

Per-cycle Film Thickness is not a Result-Effective Variable

Applicant again notes that the Examiner has not pointed to any particular passage of Dillon that discloses variations in thickness above a monolayer. The Examiner again found that "Dillon et al. discloses that the thickness of an aluminum oxide layer after each cycle depends upon the amount of amorphous aluminum oxide present and the reaction mechanism (see page 239-241 et seq.)." Applicants submit that Dillon fails to disclose depositing greater than one monolayer of aluminum oxide per cycle.

The Examiner further found that "the thickness of the trimethylaluminum is shown to be dependent upon reaction conditions, easily modified by one of ordinary skill in the art through routine experimentation." First, Dillon is concerned exclusively with self limiting reactions, even if the skilled artisan could vary the thickness of the aluminum oxide deposited per cycle; there is no reason to do so. Also, there is no teaching of how to vary the thickness of aluminum

oxide per cycle. Applicants respectfully disagree that the reaction conditions of Dillon could be easily modified through routine experimentation to produce more than a monolayer of aluminum oxide. In support of this point, Applicant attaches a Declaration from Suvi Haukka and a paper by Niskanen et al. entitled "Low-Temperature Deposition of Aluminum Oxide by Radical Enhanced Atomic Layer Deposition".

Niskanen reports results for radical enhanced atomic layer deposition using TMA and oxygen radicals in the temperature range of 25-300°C and compares the results to those achieved using ALD processes with TMA and water (see abstract). Niskanen found that the highest reported growth rate for a TMA-H_2O process was 0.19 nm/cycle, which is less than a monolayer. ("The growth rate at room temperature in the present process [disclosed in this case] was 0.29 nm per cycle, which is considerably higher than the highest rate of 0.19 nm/cycle reported for the TMA-H_2O process"; Niskanen et al "Low-Temperature Deposition of Aluminum Oxide by Radical Enhanced Atomic Layer Deposition" Journal of The Electrochemical Society, page F92, 152 (7) F90-F93; 2005). If the per cycle film thickness could be so easily modified through routine experimentation, as found by the Examiner, then it would have been done in order to achieve a growth rate of greater than one monolayer per cycle with the TMA-H_2O ALD process. According, to Niskanen et al. this had not been accomplished (other than with the claimed process) as of the publication of the attached paper in 2005.

Further, only result effective variables can be optimized. M.P.E.P. § 2144.05 II B. A particular parameter must first be recognized as a result-effective variable, i.e., a variable which achieves a recognized result, before the determination of the optimum or workable ranges of said variable might be characterized as routine experimentation. *In re Antonie*, 559 F.2d 618, 195 USPQ 6 (CCPA 1977)(The claimed wastewater treatment device had a tank volume to contractor area of 0.12 gal./sq. feet. The prior art did not recognize that treatment capacity is a function of the tank volume to contractor ratio, and therefore the parameter optimized was not recognized in the in art to be a result-effective variable.).

Per-cycle film thickness in the process of Dillon is clearly not a result effective variable. *See* Declaration of Suvi Haukka, paragraphs 5-12. Dillon is a self limiting process. The skilled artisan would appreciate that modifying the process conditions, such as temperature, reactants pulse length, reactor pressure, etc. would not be expected to deposit more than one monolayer of

aluminum oxide per cycle. Declaration of Suvi Haukka, paragraphs 5-12. The thickness of the deposited layers cannot be optimized in ALD processes because ALD is a self limiting process.

The Examiner also found that "[a]nyone of ordinary skill in the art would recognize that the thickness of a deposited film, then, is a function of the time of substrate exposure to the precursor." (Office Action at page 3). This is incorrect because supplying a greater amount of TMA or water would not increase the deposition of aluminum oxide to above one monolayer per cycle in Dillon's ALD process. Declaration of Suvi Haukka, paragraph 10. Accordingly, Applicant respectfully requests withdrawal of this rejection for at least this reason.

Further, as argued in previous responses, this point is plainly supported by Dillon because Dillon's reactions are explicitly self-limited. *See, e.g., Dillon,* at abstract ("both the (A) and (B) reactions were ***self-limiting*** and complete"); *id.*, at 231 ("***self-limiting*** surface reactions were observed..."); *id.*, at 238 ("spectra indicate that both reactions are complete and ***self-limiting***").

Applicant again notes that Dillon does not disclose variations in the per-cycle thickness of a deposited layer, and certainly does not disclose variation above a full monolayer as claimed. Dillon's disclosed reactions are each clearly self-limited to no more than a monolayer, and Dillon describes them as such. Further, while Dillon does not measure the growth rate per cycle, Dillon acknowledges that similar reaction conditions resulted in growth of only 0.33 ML of aluminum oxide per cycle. ("Because the cross section of the amorphous Al_2O_3 infrared absorbance is not known, the Al_2O_3 deposition per AB cycle can not be determined from these results. However, earlier work has indicated that ***0.33 ML of Al_2O_3 is deposited per AB reaction cycle under similar conditions***". Dillon, page 241.)

In conclusion, per-cycle film thickness in the process of Dillon is not a result effective variable. Declaration of Suvi Haukka, paragraphs 5-12. Contrary to the Examiner's findings, this is plainly supported by Dillon, the art of record, the attached paper by Niskanen et al., and the Declaration of Suvi Haukka. Accordingly, Applicant respectfully requests withdrawal of this rejection.

The Combination Proposed by the Examiner fails to Make Claims 1-4 Obvious

The prior art reference (or references when combined) must teach or suggest all of the claim limitations: "the need to demonstrate the presence of all claim limitations in the prior art

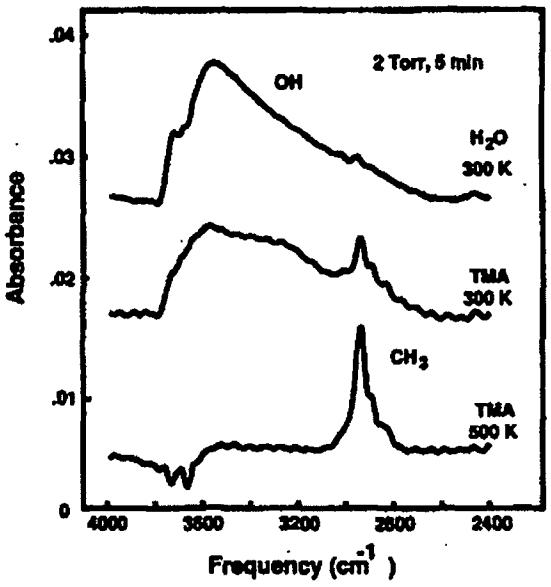
was not obviated [by KSR]", *Abbott Labs. v. Sandoz, Inc.*, 2007 WL 1549498, *4 (N.D. Ill. May 24, 2007). Penneck fails to make up for the deficiencies of Dillon noted above.

The Examiner again states "though the applicant argues that the process of Dillon is self-limiting, Dillon discloses using the same precursor as the applicant, so if the use of this precursor was truly self-limiting, the 'more than one monolayer' limitation in claim 1 would be improper." Just because two processes use the same reactant does not mean that they will achieve the same result. Moreover, it is not the nature of the TMA precursor in Dillon that leads to the self-limiting nature of the reaction but rather the ***deposition conditions***. In Dillon, the deposition conditions are such that TMA adsorbs in a self-limiting manner. In contrast, in the claimed process, the deposition conditions are such that more than one monolayer of TMA forms on the substrate surface per cycle. These deposition conditions are necessarily different from those in Dillon. Thus, while Applicant's claimed process and Dillon's process both use TMA, the processes are different, and thus the same results would not be expected. The combination proposed by the Examiner fails to disclose "exposing the substrate in the reaction chamber to gaseous trimethyl aluminum (TMA), such that more than one monolayer of TMA forms on the substrate surface". Thus, Applicants respectfully request withdrawal of this rejection for at least this reason.

Applicant points out that, ***under the process conditions disclosed by Dillon, including*** the particular temperatures and pressures, TMA adsorbs on the substrate in a self-limiting manner and will not deposit more than one monolayer per cycle, as explicitly recognized by Dillon itself. Applicant, on the other hand, discloses and claims formation of more than one monolayer of TMA on the substrate per cycle. This can be achieved by using ***different*** process conditions than those that are used in Dillon or typical ALD, namely those conditions that would cause more than one monolayer of TMA to form on the surface of the film. Because more than one monolayer of Al_2O_3 cannot be formed unless multiple monolayers of TMA are formed, the combination proposed by the Examiner also fails to disclose "wherein in each cycle more than one monolayer of Al_2O_3 is formed." While Dillon does not measure the growth rate per cycle, Dillon acknowledges that similar reaction conditions resulted in growth of only 0.33 ML of aluminum oxide per cycle. ("Because the cross section of the amorphous Al_2O_3 infrared absorbance is not known, the Al_2O_3 deposition per AB cycle can not be determined from these

results. However, earlier work has indicated that ***0.33 ML of Al_2O_3 is deposited per AB reaction cycle under similar conditions***". Dillon, page 241.). Thus, Applicant requests withdrawal of this rejection for at least this reason.

Claim 4 additionally recites carrying out the process of Claim 1 at room temperature. Dillon discloses ***annealing*** studies performed at room temperature, and Dillon performs an experimental ***non-cyclical*** chemisorption of TMA at 300K combined with a ***subsequent*** exposure at 500K, but Dillon does not disclose an **ABAB** reaction in which the **A** reaction (much less the entirety of the reaction) is performed at room temperature. In fact, if anything, Dillon implies that chemisorption of TMA at 300K is not useful in an **ABAB** process. *See* Dillon, at 232-33 (two consecutive 5-minute exposures of alumina membrane to TMA at 300K left broad absorbance of O-H stretching vibrations, indicating substantially incomplete reaction); *see also* id., at Fig. 2 below (showing incomplete reaction at 300K and completion at 500K); id., at Fig. 3 (performing sequential exposures to TMA at 500K, but not at 300K); id., at 238 ("TMA reaction with a hydroxylated alumina surface does not go to completion at 300K"). Nowhere does Dillon disclose or suggest that an exposure to TMA at 300K is possible or useful in a sequential process. Accordingly, Applicants respectfully request withdrawal of the rejection of Claim 4 for at least this reason.



No Reasonable Expectation of Success

Moreover, one skilled in the art would have no reasonable expectation of success. *Pharmastem Therapeutics v. Viacell, Inc.* 491 F.3d 1342, 83 U.S.P.Q.2d 1289 (Fed. Cir. 2007) (after *KSR*, Federal Circuit finds claims non-obvious for lack of indication of reasonable expectation of success for asserted combination). Further, "[t]o the extent that an art is unpredictable, as the chemical arts often are, *KSR*'s focus on these 'identified, predictable solutions' may present a difficult hurdle because potential solutions are less likely to be genuinely predictable." *Eisai Co. Ltd. v. Dr. Reddy's Laboratories, Ltd*, Nos. 2007-1397, -1398, slip opinion at pg 8 (Fed Cir. 2008).

As discussed above, Dillon only discloses a self limiting process and provides no teaching or suggestion of how to form more than one monolayer of TMA per cycle. Thus, the skilled artisan would have no reasonable expectation of success for a process in which "more than one monolayer of TMA forms on the substrate surface" or "wherein in each cycle more than one monolayer of Al_2O_3 is formed." Accordingly, Applicant respectfully requests withdrawal of this rejection for at least this reason.

No Disclaimers or Disavowals

Although the present communication may include alterations to the application or claims, or characterizations of claim scope or referenced art, Applicant is not conceding in this application that previously pending claims are not patentable over the cited references. Rather, any alterations or characterizations are being made to facilitate expeditious prosecution of this application. Applicant reserves the right to pursue at a later date any previously pending or other broader or narrower claims that capture any subject matter supported by the present disclosure, including subject matter found to be specifically disclaimed herein or by any prior prosecution. Accordingly, reviewers of this or any parent, child or related prosecution history shall not reasonably infer that Applicant has made any disclaimers or disavowals of any subject matter supported by the present application.

Please charge any additional fees, including any fees for additional extension of time, or credit overpayment to Deposit Account No. 11-1410.

Respectfully submitted,

KNOBBE, MARTENS, OLSON & BEAR, LLP

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Low-Temperature Deposition of Aluminum Oxide by Radical Enhanced Atomic Layer Deposition

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Aluminum oxide was deposited by radical enhanced atomic layer deposition using trimethylaluminum (TMA) and oxygen radicals in the temperature range 25-300°C. The radicals were produced by dissociating oxygen gas in a remote microwave plasma discharge. Oxygen was mixed with argon which was also used as the carrier and purge gas. Films were grown on silicon, glass, and indium tin oxide coated glass substrates. Additional growth experiments were conducted on heat-sensitive materials: polyethene, polypropene, and wool. The time to complete one deposition cycle was nearly independent of the deposition temperature, being around 10 s for all deposition temperatures. Growth rates were between 1.5 and 2.9 Å per cycle, which is higher than what has been obtained with the TMA-H_2O process in similar reactor conditions. The films were amorphous according to X-ray diffraction. The films were also very smooth; the surface root-mean-square roughness was less than 0.8 nm for 180 nm thick films. The films had breakdown fields, defined as the field corresponding to the leakage current density of 1 $\mu A/cm^2$, between 6 and 10 MV/cm, and dielectric constants between 6.5 and 8.1. The film impurity levels according to time-of-flight elastic recoil detection analysis were between 0.8 and 15 atom % for hydrogen and 0.2 and 4 atom % for carbon. The refractive indexes at 580 nm were between 1.60 and 1.64.
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Aluminum oxide thin films have many applications, as Al_2O_3 can be used as a dielectric, passivating, and protecting material. In electroluminescent thin-film displays aluminum oxide has all these roles. It is used as a passivation layer on glass to prevent sodium diffusion from glass to the thin-film structure, as a dielectric layer on top and bottom of the luminescent layer, and finally, an Al_2O_3 layer encapsulates the whole thin-film structure.[1-4] Aluminum oxide is also known as a wear-resistant coating material and recently it has demonstrated excellent behavior in microelectromechanical devices.[5] Yet another new application for thin aluminum oxide films is as a thin-film magnetic head gap layer.[6] Aluminum oxide film can also improve the gas permeation properties of polymers.[7]

In microelectronics new high-k dielectric oxide materials to replace silicon dioxide have been extensively studied.[8,9] Aluminum oxide has good electrical properties such as wide bandgap (8.7 eV) and high electric field strength (8 MV/cm). The dielectric constant, however (k = 9), is only double to SiO_2.[6,8,10] Al_2O_3 is stable on bare silicon, which makes it an attractive material for gate dielectrics in metal-oxide-semiconductor (MOS) transistors.[11-14] Because of the stability, Al_2O_3 can be deposited on bare silicon without the formation of a capacitance degrading interfacial SiO_{2-x} layer.[13,15] An interfacial SiO_{2-x} layer may be formed, however, by extrinsic reasons, such as oxidation by atmospheric oxygen after the deposition or by a strong oxidant during the deposition.[13,14] If the moderate k-value limits the use of pure Al_2O_3 as gate oxide, it can be a component in a multilayer structure or solid solution with Zr, Hf, Ta, or rare earth oxides, for example.[16-19] Also, aluminate compounds, such as $LaAlO_3$, have been considered potential high-k materials.[20]

Atomic layer deposition (ALD) is an attractive thin-film deposition technique because it allows conformal deposition on structured surfaces.[1,21] Many of the applications mentioned above require conformal films. Al_2O_3 has been deposited by ALD with many processes from which the one using trimethylaluminum (TMA) and water has been the most extensively studied.[22-24] This is probably the most successful ALD reaction and it operates at a very broad temperature range (30 - 500°C).[7,24] Alternatively, ozone and direct oxygen plasma have been used as an oxygen source in ALD of Al_2O_3 from TMA,[25-27] and in one case the oxygen source has been aluminum metal oxide.[15] Stable thin-film growth can be achieved by the TMA-H_2O process down to 30°C and the growth rate per cycle is higher than at elevated temperatures. However, the films grown at 100°C or less contain significant amounts of hydrogen.[7] It appears that the composition of the low-temperature films is close to AlOOH and they may even contain $Al(OH)_3$. Furthermore, the purging of water is very slow close to room temperature due to its polar nature. At 30°C deposition temperature the time required to purge water was as long as 180 s, which was almost 90% of the total ALD cycle time.[7] The slow purging of water makes the effective deposition rate low and can thus limit the feasibility of a process in potential applications.

In this paper Al_2O_3 films are grown by ALD at low temperatures using oxygen radicals obtained from a remote plasma discharge. This paper aims to circumvent three problems of the known low-temperature Al_2O_3 processes: (*i*) avoid possible substrate damage by energetic particle bombardment by using remote plasma, (*ii*) reduce the hydrogen content and (*iii*) decrease the cycle time by replacing water with oxygen radicals. The goal is to grow protective films on temperature-sensitive substrates like polymers and wool, and obtain a fast process with good film qualities even at low deposition temperatures.

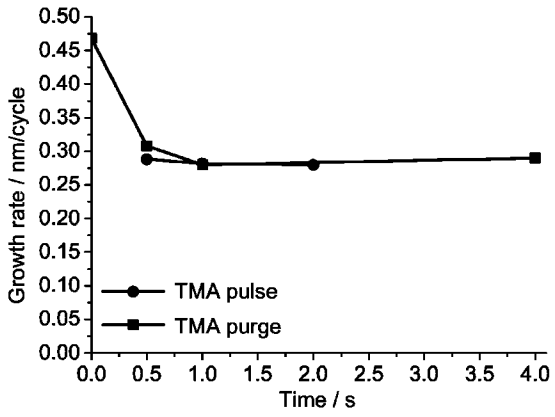

Figure 1. Growth rate dependence on the TMA pulse length and the following purge time at room temperature, 25°C. The pulse time experiments were done with a purge time of at least four times the pulse time. The purge time experiments were done with a 0.5 s TMA pulse.

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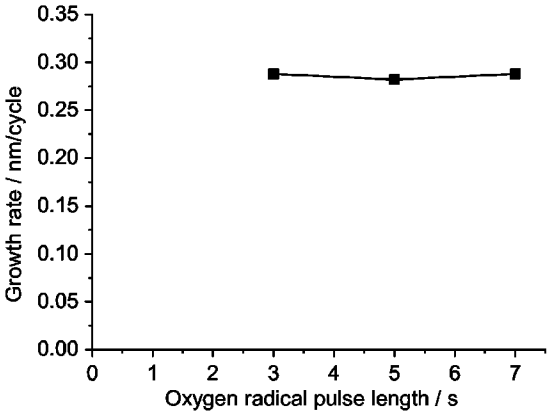

Figure 2. Growth rate dependence on the oxygen pulse length at room temperature, 25°C. The experiments were conducted using 0.5 s TMA pulse with 1 s purge period.

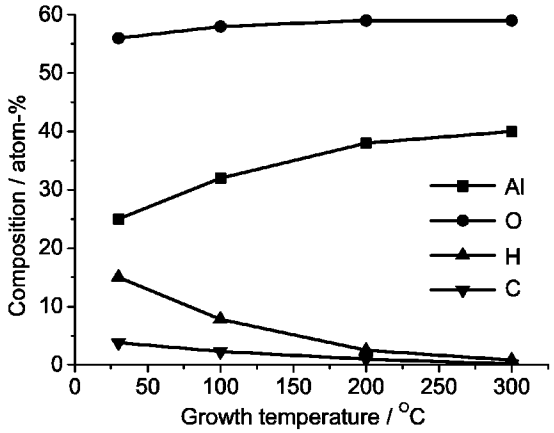

Figure 4. Film composition according to TOF-ERDA as a function of deposition temperature.

Experimental

The growth experiments were carried out in a flow-type ALD reactor with inert gas valving.[1] The reactor had been modified to incorporate a remote microwave plasma source for radical generation.[28] The overall carrier and purge gas was argon (AGA, −99.98%) which was purified with Aeronex GateKeeper inert gas purifier to decrease the impurity levels below 1 ppb. TMA (Witco) was used as the aluminum precursor and it was kept at room temperature, 23°C. Oxygen radicals, produced from molecular oxygen gas (AGA, 99.999%) by the plasma discharge, were used as the oxygen source. Aluminum oxide was grown on several substrate materials: n-type silicon, borosilicate glass, and indium tin oxide (SnO_2:In, ITO) coated glass. Also, some highly heat-sensitive substrate materials were used: polyethene, polypropene, and wool.

Film crystallinities were studied with grazing incidence X-ray diffraction (GIXRD). Film density and interface roughness were measured using X-ray reflectivity (XRR). Both measurements were conducted with a Bruker-axs D8 Advance diffractometer/reflectometer operated in parallel beam geometry. For some samples, also the film thickness was determined with XRR measurements. For the majority of samples either the film thickness was too large or the density difference between the film and the substrate was too small, and the XRR curves could not be modeled. Film thicknesses and refractive indexes were determined by fitting optical reflectance spectra measured within a wavelength range of 370 - 1100 nm using a Hitachi U-2000 spectrophotometer.[29]

To measure leakage current densities and capacitances of the films, aluminum oxide films were grown on sputter-deposited ITO films at four different temperatures ranging from room temperature to 300°C. The top electrodes were aluminum dots, evaporated through a shadow mask. The evaporation was done with an Instrumentti Mattila IM-1992 electron-beam evaporator using aluminum pellets (99.99% Al, Cerac) as the source material. A Keithley 2400 SourceMeter was used for measuring the leakage current densities. Capacitance measurements were done with an HP4284A LCR meter using a 100 kHz measuring frequency. The breakdown voltage and dielectric constant were measured from several electrodes, and the reported values were obtained from at least three electrodes.

The impurity content of the films was analyzed by time-of-flight elastic recoil detection analysis (TOF-ERDA).[30] The presence of Al_2O_3 on the polymers and wool was verified by scanning electron microscopy (SEM) using a Zeiss DSM 962 electron microscope, and energy-dispersive X-ray spectroscopy (EDX) using a Link ISIS spectrometer. Fourier-transform infrared spectroscopy (FTIR) performed with a Perkin Elmer Spectrum GX spectrophotometer in transmission mode was used to study the aluminum oxide phases and the chemical nature of the impurities.

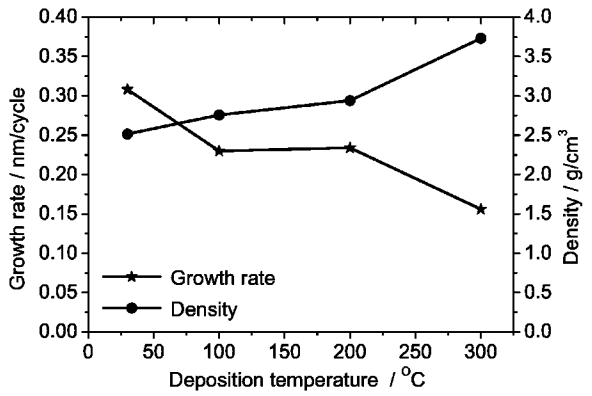

Figure 3. Growth rate and film density dependence on the deposition temperature.

Results and Discussion

Al_2O_3 films could be grown on all substrate materials tested. The growth was successful on even the most sensitive polymer and wool without damaging the substrates. The TMA source length was varied to study its effect on the film growth rate (Fig. 1) at room temperature. The saturation of growth rate occurred with TMA pulse length of 0.5 s. The purge period following the TMA pulse had to be at least of equal length with the TMA pulse, otherwise some growth occurred in chemical vapor deposition (CVD) mode (Fig. 1). The growth rate at room temperature saturated even with a 3 s oxygen radical pulse (Fig. 2). This is the shortest controlled oxygen radical pulse time that can be obtained, because the discharge column extends slowly from the microwave plasma source.[28,31] Increasing the oxygen pulse length increases the area of saturated growth, because the source is point-like. The 5 s pulse time used in the majority of experiments already results in large enough diameter (about 3 cm)

Table I. Film composition according to TOF-ERDA at four different deposition temperatures.

Deposition temperature (°C)	Film composition (atom %)			
	Al	O	H	C
25	25	56	15	3.8
100	32	58	7.8	2.3
200	38	59	2.5	1
300	40	59	0.8	0.15

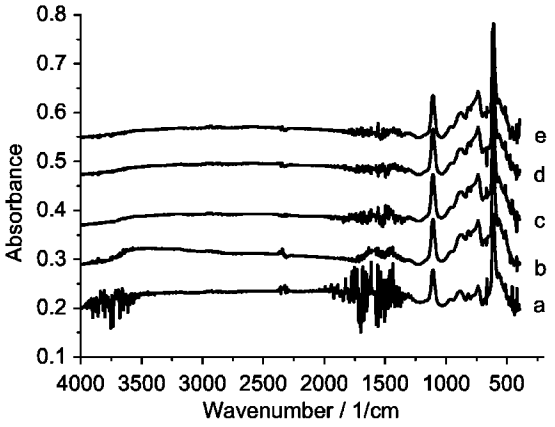

Figure 5. Infrared absorption spectra of aluminum oxide films grown utilizing (a) H_2O and (b-e) O-radicals as the oxygen precursor. The film deposition temperatures were (a) 150, (b) 25, (c) 100, (d) 200, and (e) 300°C.

for easy characterization of the film properties. The purge period following the oxygen pulse was 3 s, which is required to allow the flow through the plasma source to stop. The dependence of saturated film growth rate on substrate temperature was also studied (Fig. 3).

The time required to complete one ALD cycle at room temperature was 10 s, whereas the cycle time for TMA-H_2O process at room temperature was about 200 s, largely because of the need for a long, 180 s purge period after the water pulse.[7] The growth rate at room temperature in the present process was 0.29 nm per cycle, which is considerably higher than the highest rate of 0.19 nm/cycle reported for the TMA-H_2O process.[32] The growth rate decreased with increasing deposition temperature: at 100 and 200°C the growth rate was 0.23 nm/cycle and at 300°C, 0.15 nm/cycle. The decreasing growth rate with increasing growth temperature can be somewhat attributed to increasing density (Fig. 3). The thickness values obtained with UV-visible (UV-vis) and XRR measurements agree very well: the differences between the values obtained by these two techniques were less than 5%. Refractive indexes obtained by UV-vis measurements were between 1.60 and 1.64 at 580 nm wavelength for samples grown at 25 and 300°C, respectively. The values are similar to what has been obtained with the TMA-H_2O process.[24]

According to XRD the as-deposited films were amorphous, which is an expected result for Al_2O_3 grown by ALD. The films were smooth according to the XRR measurements: the surface root mean square (rms) roughness values were below 0.8 nm for about 180 nm thick films.

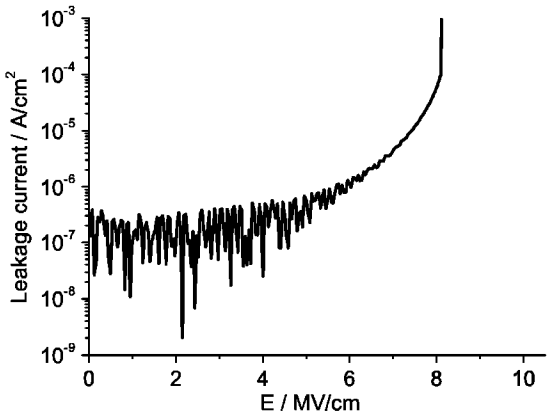

Figure 6. Leakage current *vs.* electric field for Al_2O_3 grown at room temperature, 25°C.

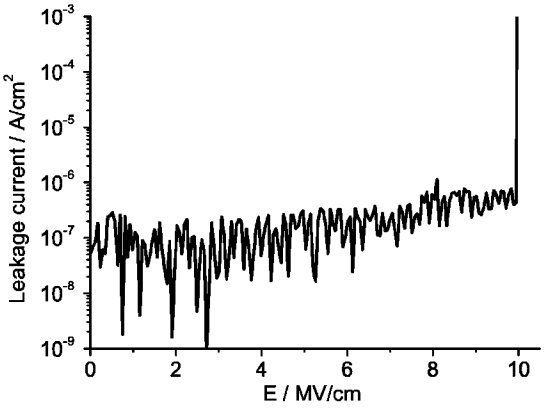

Figure 7. Leakage current *vs.* electric field for Al_2O_3 grown at 300°C.

The impurity contents of the films decreased steadily with increasing temperature and the impurities were exclusively carbon and hydrogen (Fig. 4). Based on the film composition (Table I), the film grown at 25°C would seem to be close to AlOOH. Films grown at 100°C and above are more clearly Al_2O_3 but with CH_3 impurities. However, based on the FTIR measurements (Fig. 5), all samples were similar, even the one grown at 25°C, and contained only features associated to Al_2O_3 and not AlOOH.[33] The only difference between the samples is in the intensity of the very broad AlO-H stretch between 2600 and 3800 cm^{-1},[34] which increases with decreasing deposition temperature. Thus, as the sample grown at 300°C is nearly pure Al_2O_3 (Table I) and the sample grown at 25°C displays similar FTIR spectrum, the latter seems to be Al_2O_3 also. The exact chemical nature of the impurities in the sample grown at 25°C, however, remains unclear.

As a general trend, the leakage current densities decreased with increasing deposition temperature as did the breakdown fields (Fig. 6-8). Breakdown field was defined as the field causing a 1 $\mu A/cm^2$ current density. No clear trend could be observed, however, for the behavior of the dielectric constant (Fig. 8). For the sample grown at room temperature the breakdown field was 5.8 MV/cm and the dielectric constant, ε, was 7.4. For the sample grown at 100°C the breakdown field increased to 9.2 MV/cm but the dielectric constant was only 6.5. For the sample grown at 200°C the breakdown field did not change much, being 9.5 MV/cm, but the dielectric constant increased to 8.1. Finally, for the sample grown at 300°C both values remained nearly the same: the breakdown field increased slightly to 10 MV/cm and dielectric constant was 7.8.

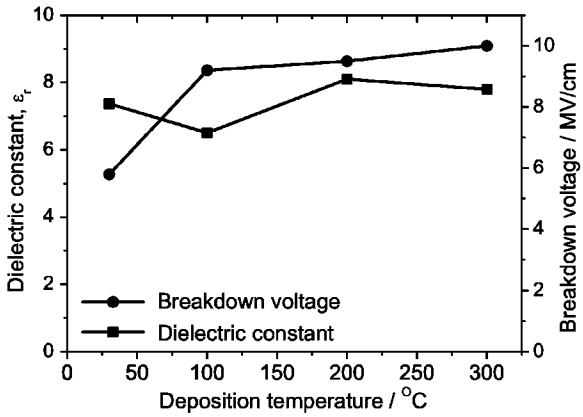

Figure 8. Breakdown voltage and dielectric constant as a function of deposition temperature. The breakdown voltage values are within an error range of 5% and dielectric constant 1%.

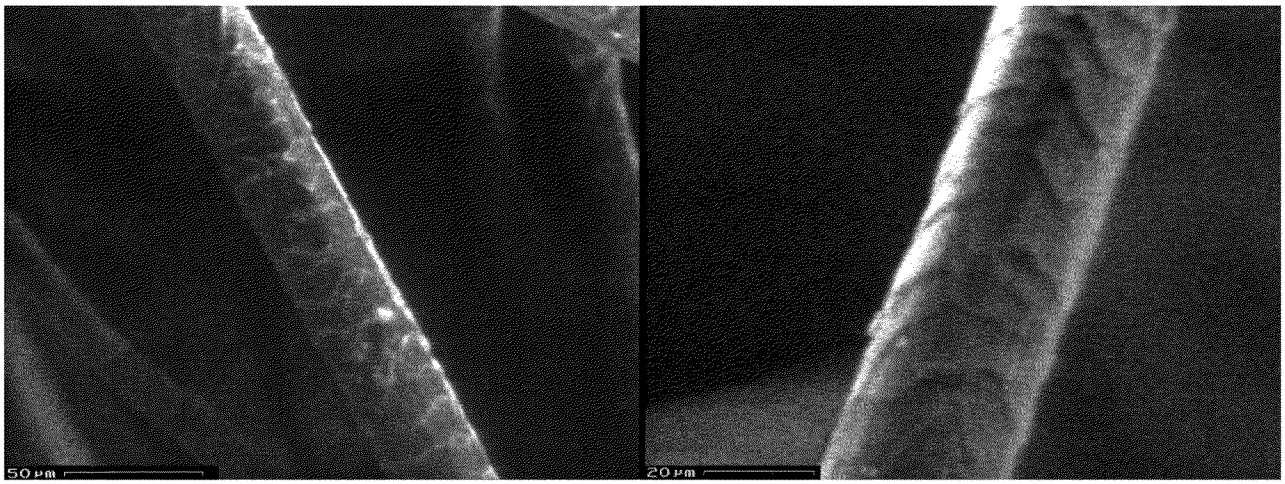

Figure 9. SEM images of wool before and after deposition. The left image is the fiber without Al_2O_3 at 500 times magnification, and on the right is the one with Al_2O_3 at 1000 times magnification.

The film growth on polymers and wool was verified by EDX analysis: all polymers, including two different types of Teflon substrates and wool, exhibited strong signals from aluminum whereas the nondeposited reference substrates did not. Additionally, EDX measurements gave an upper limit for the growth rate of Al_2O_3 on polyethene and Teflon. The growth rate was slightly higher than on silicon, but this may be explained by porosity or roughness of the substrate. Nevertheless, EDX measurements confirm at least qualitatively that the growth rate is more or less the same on polyethene and Teflon as on silicon. SEM images revealed that the most sensitive substrate material used, wool, exhibited very similar surface features before and after deposition (Fig. 9). This indicates that the process is gentle enough even if it uses oxygen radicals as the oxygen source. In related studies done in our laboratory, it was found that wool burns almost instantly in oxygen plasma but can withstand some exposure to oxygen radicals. In this light, it seems that the aluminum oxide being deposited protects the wool beginning already from the first deposition cycles.

Conclusions

Al_2O_3 could be successfully grown at room temperature on several substrate materials including heat-sensitive polymers and wool. The cycle times at room temperature were very fast compared to the TMA-H_2O process, and further improvement may be expected with a different reactor plasma source design. Also, the electrical properties were already good for the films grown at room temperature and improved steadily with increasing growth temperature with the best values obtained at 300°C. The successful growth at room temperature on polymers without destroying them makes this process interesting for gas permeation application, for example. The hydrogen content is high for films deposited at room temperature but is still much lower than in the TMA-H_2O process at 33°C and up to deposition temperatures close to 100°C.

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